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**LMH-1/HYDRAZINE HETEROGENEOUS  
PROPELLANT DEVELOPMENT**

**E. T. Niles, B. H. Seaman and E. J. Wilson**

**QUARTERLY PROGRESS REPORT AFRPL-TR-67-225  
(1 MAY TO 31 JULY 1967)**

**AUGUST 1967**

**AIR FORCE ROCKET PROPULSION LABORATORY  
RESEARCH AND TECHNOLOGY DIVISION  
EDWARDS AIR FORCE BASE, CALIFORNIA**

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**(Prepared under Contract Nr. AF 04(611)-11606 by  
The Dow Chemical Company,  
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**FOREWORD**

This report was prepared by The Dow Chemical Company, Midland, Michigan, under USAF Contract Nr. AF 04(611)-11606. The contract was initiated under Air Force BPSN Nr. 623148, AFSC Project No. 3148, "LMH-1/Hydrazine Heterogeneous Propellant Development." The work was administered under the direction of the Rocket Propulsion Laboratory, Edwards Air Force Base, With Mr. G. Allen Beale as Air Force Project Officer.

This is the fifth quarterly report, covering the work performed during the period of 1 May 1967 through 31 July 1967. The Dow Report Number is HZ-5Q-67.

Management direction at Dow is under Dr. R. P. Ruh, Laboratory Director, and the work is supervised by Dr. D. A. Rausch, Assistant Laboratory Director. Dr. E. T. Niles is the principal investigator with Mr. E. Wilson and Mrs. B. H. Seaman making major contributions.

Publication of this report does not constitute Air Force approval of report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

G. Allen Beale  
Project Engineer, AFRPL

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## (U) ABSTRACT

(C) Research directed toward the development of a stable, storable, heterogeneous  $\text{AlH}_3$ -1451/ $\text{N}_2\text{H}_4$  propellant with desirable rheological properties is being conducted on an integrated program to furnish a gel system and to study the compatibility of the system components. Phase I, the development and selection of a system, has been completed in this quarter and a cross-linked polyacrylamide gellant has been selected from the four thickening agent candidates. The selected system will be evaluated and characterized in Phase II. The selection of this polyacrylamide system was based on its superior physical stability characteristics, since all of the candidate systems had adequate physical properties. Recent work has been concentrated on the polyacrylamide gelling agent and heterogeneous systems based on this agent. In addition, the study of the system components is being continued.

(C) Work is being conducted to determine the chemical nature of the polyacrylamide agent as related to water, hydrazine, and strong bases, such as tetramethylguanidine. The effect of aging, at ambient temperature and at  $60^\circ\text{C}$ ., on the gel physical properties also is being studied.

(C) The effect of the polyacrylamide gelling agent on the stability of aluminum hydride-1451/ $\text{N}_2\text{H}_4$  formulations was determined. An increase in the initial gas evolution was noted in the gelled sample, as compared to a reference sample containing no gelling agent, but the long-term stability of the gelled sample was at least equivalent to that of the reference sample.

(C) Aluminum hydride was found to be stabilized in hydrazine after an initial reaction of the two components. A decelerating rate of gas evolution is established for an indefinite length of time, if the hydride is left in contact with the hydrazine. If the hydride is isolated and reformulated with propellant grade hydrazine, much less gas is generated initially and the long-term stability persists. The gas generation of hydride samples (neat and in hydrazine) which have been pretreated with a small amount of water in hydrazine and in other solvents is being observed at  $60^\circ\text{C}$ . The greatest reduction of initial gas evolution was achieved from a prolonged hydrazine pretreatment at  $60^\circ\text{C}$ . Equivalent stabilization of the aluminum hydride-1451 appears to be possible with n-butylamine and ethyl alcohol in less time than the hydrazine treatment, as indicated by preliminary results from the screening of various treatment liquids.

(C) Another effect of hydrazine upon the stability of aluminum hydride-1451 was noted. Hydrazine essentially stopped the rapidly accelerating hydride decomposition.

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(C) Additional studies are being conducted to specify the requirements of aluminum hydride for future propellant scale-up preparation. Previously, Mg-doped DPA treated hydride had been selected as the most stable type of an aluminum hydride-1451/hydrazine propellant. Studies of additional lots of Mg-doped, DPA treated hydride have shown large variations among hydride samples in the initial stability of hydride/hydrazine formulations which do not correlate with either the stability of the neat hydride or common impurities detected by analysis. The effects of crystal size and surface phenomenon are discussed.

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SECTION I(U) TECHNICAL RESULTS AND DISCUSSION

(C) Research directed toward the development of a stable, storable, heterogeneous  $\text{AlH}_3\text{-1451/N}_2\text{H}_4$  propellant with desirable rheological properties is being conducted on an integrated program to furnish a gel system and to study the compatibility of the system components. Phase I, the development and selection of a system, has been completed in this quarter and work has been started on the evaluation of the selected system, which is Phase II of this program. The research is conveniently divided into two areas for the purpose of discussion: (i) polyacrylamide gels and (ii) stability studies.

A. POLYACRYLAMIDE GELS (U)

(C) During this period a crosslinked polyacrylamide was selected from the four thickening agent candidates for further evaluation and system characterization in Phase II of this program. The four systems based on (i) polyacrylamides, (ii) acrylic acid/acrylamide copolymers, (iii) ammonium polyacrylates, and (iv) OSH emulsifier\* all had adequate physical properties. The polymeric gelled systems were cohesive with desirable rheological properties while the OSH emulsion system was adhesive with good rheological properties. The selection of the polyacrylamide system was based on its superior physical stability characteristics as shown in accelerated aging tests and its relative stability in the atmosphere. Thus, work during this quarter has been concentrated on the polyacrylamide gelling agent and heterogeneous systems based on this agent.

1. Homogeneous Gels (U)

(U) With the selection of the polyacrylamide gelling agent, a study was undertaken to determine its chemical nature as related to hydrazine gels. Polyacrylamides can be prepared with

varying degrees of hydrolysis, i.e., some of the  $\begin{array}{c} \text{O} \\ || \\ -\text{C}-\text{NH}_2 \end{array}$  groups

are hydrolyzed to  $\begin{array}{c} \text{O} \\ || \\ -\text{C}-\text{OH} \end{array}$ . Early in the program it was found that a low degree of hydrolysis was desirable, and the majority of the work was conducted with polyacrylamide which were about 0.05% hydrolyzed. It is known that water gels prepared with polyacrylamides can undergo some breakdown under slightly acid conditions; they are stabilized under basic conditions. Thus, hydrazine should afford a stabilizing medium for polyacrylamides.

(U) To determine the effect of water and hydrazine on the agent, gels containing 2% polyacrylamide were prepared and subjected to 40°C. and 70°C. surveillance tests. The yield stress of the gels was determined over a period of 48 hours. The

\*The reaction product of oleylsarcosine and hydrazine.

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change in yield stress of the hydrazine gel was not significant, but some breakdown was indicated in the water gel after 24 hours at 70°C. These data are presented in Table I.

Table I

(U) The Effect of Time and Temperature  
on Yield Stress of Homogeneous 2% Polyacrylamide  
Water And Hydrazine Gels

Time hr.	Yield Stress, dynes/cm. <sup>2</sup>			
	40°C.		70°C.	
	<u>N<sub>2</sub>H<sub>4</sub></u>	<u>H<sub>2</sub>O</u>	<u>N<sub>2</sub>H<sub>4</sub></u>	<u>H<sub>2</sub>O</u>
0	583	335	583	335
2	344	244	298	249
4	326	308	293	363
24	396	258	265	57
48	391	233	407	186

(U) In another experiment, a strong base (tetramethylguanidine) was added to the hydrazine from which a homogeneous gel was prepared. The yield stress of the gel increased markedly after 48 hours at 70°C. and, also, after 72 hours at 40°C., as indicated in Table II.

Table II

(U) Effect of Tetramethylguanidine on Yield Stress  
of a Homogeneous 2% Polyacrylamide Hydrazine Gel

Time hr.	Yield Stress, dynes/cm. <sup>2</sup>	
	<u>40°C.</u>	<u>70°C.</u>
0	383	383
2	336	266
4	327	370
24	392	289
48	370	730
72	660	1801

The reason for this increase is unknown and is the subject of further investigation. In addition, other samples of polyacrylamide have been prepared with about 0.5% hydrolysis and they are being investigated in a similar manner.

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**2. Heterogeneous Gels (U)****a. Physical Integrity (U)**

(C) Long-term stability tests are being conducted on heterogeneous  $\text{AlH}_3$ -1451/hydrazine (40:60) propellants to detect changes in physical properties during storage. One propellant sample with 0.5% polyacrylamide has not undergone a significant change after 42 days at ambient temperature. In an accelerated test at 60°C., the same sample had a sharp increase in yield stress after seven days. Another propellant sample, prepared with 0.42% polyacrylamide is unchanged after 70 days at ambient temperature. These studies will be continued and various types of hydride and treated hydride will also be studied in this manner.

**b. Chemical Stability (U)**

(C) Three gelling agents and one emulsifier have been studied previously for their effect on the chemical stability of  $\text{AlH}_3$ -1451/ $\text{N}_2\text{H}_4$  propellant systems (1). During this quarter the stability of the complete gelled system with the polyacrylamide agent was evaluated.

(C) The gelled and emulsified propellants tested previously had characteristically evolved more gas initially than an ungelled reference mixture of  $\text{AlH}_3$ -1451/ $\text{N}_2\text{H}_4$  (1). The polyacrylamide gel also exhibits this increase in initial gassing as shown in Figure 1 where the gas generation of the gelled propellant is plotted together with an ungelled  $\text{AlH}_3$ -1451/ $\text{N}_2\text{H}_4$  mixture (09236 5-15-67). However, the gas generation rate of the polyacrylamide gel shows a greater tendency to decrease with time than any other gel or emulsion studied. It appears the long-term stability of the polyacrylamide gel may be superior to the  $\text{AlH}_3$ -1451/ $\text{N}_2\text{H}_4$  reference mixture.

**3. Gels With Treated Aluminum Hydride-1451 (C)**

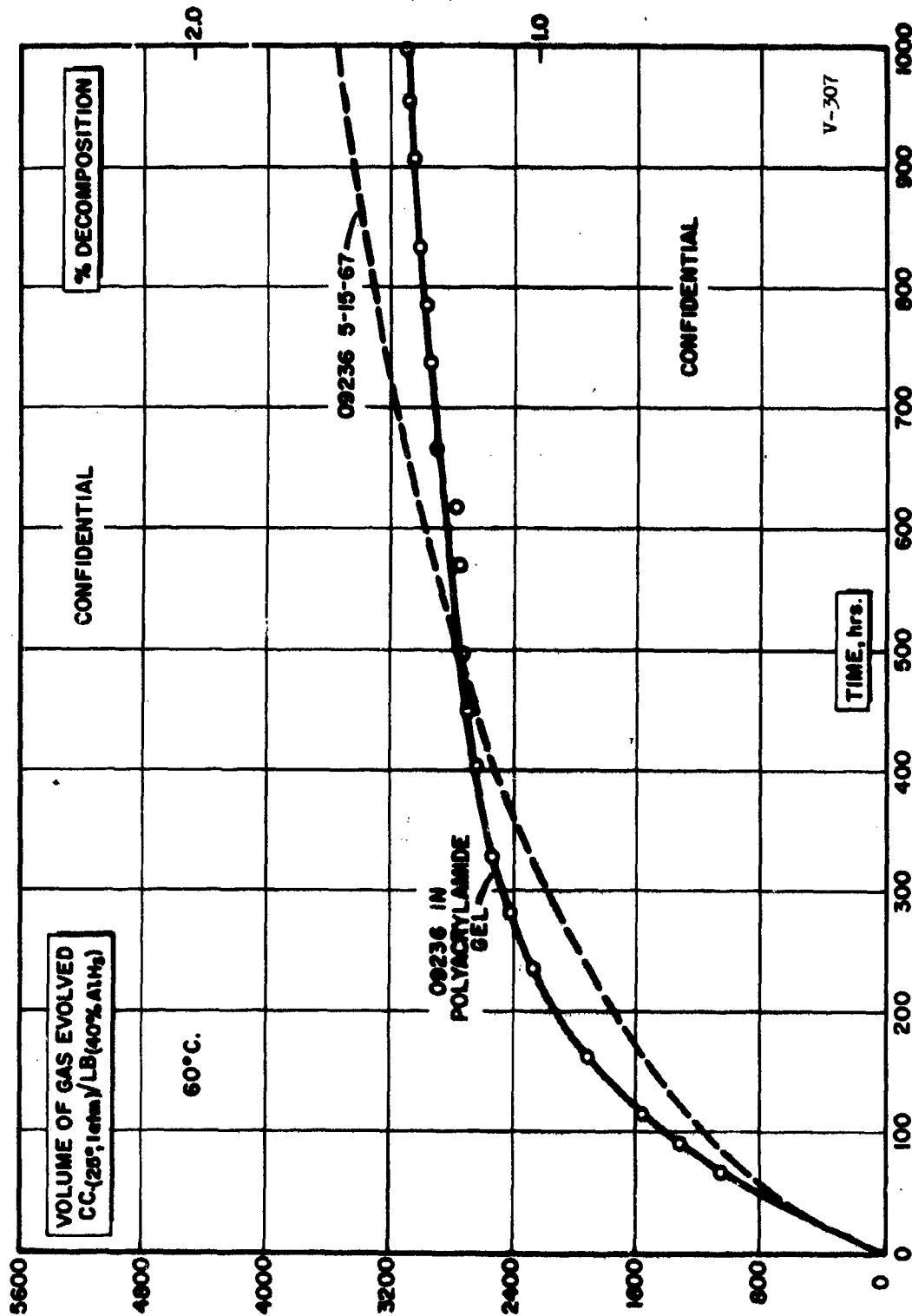
(C) Several screening gels have been made which contained pretreated  $\text{AlH}_3$ -1451 (pretreatment is more fully discussed in Section I.B.6). Most heterogeneous polyacrylamide gels that have been evaluated contained hydride which was only washed in hydrazine. Washing the hydride with hydrazine removed trace extraneous ions, such as lithium and chloride, and brought about a definite improvement in the gel physical integrity. The pretreatment of the hydride would be expected to improve the gel integrity as much as, or more than, the washing procedure, because of the longer contact time at a higher temperature, but the results were not consistent with this assumption.

(C) During the pretreatment, the hydride was in contact with the treatment liquid for six days at 60°C. It was then isolated and dried. Gels were prepared by combining the treated hydride with dry polyacrylamide gelling agent and then adding hydrazine with stirring. The gel containing hydrazine-treated hydride was very similar to the gel containing benzene-treated hydride. The treated gels were only slightly improved over those which contained

<sup>1</sup>(C) Dow Quarterly Report Nr. HZ-4Q-67, Contract Nr. AF 04(611)-11606, May 1967, AFRPL-TR-67-113.

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(C) Fig. 1 - Effect of Polyacrylamide Gelling Agent on Gas Generation of Aluminum Hydride-1451/Hydrazine Formulations

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untreated hydride; the treated gels underwent syneresis shortly after mixing. However, a gel containing n-butylamine-treated hydride had good physical integrity, even though the hydride contained 0.12% chloride after treatment (see Table III). No syneresis of the gel was observed after seven days storage in an inert atmosphere, although from previous observations 0.1% chloride had been considered deleterious to gel physical integrity.

Table III

(U) Elemental Analysis of As-Received and Treated Aluminum Hydride-1451 (Lot 09236)

<u>Element</u>	<u>Aluminum Hydride-1451 Analysis, wt. %</u>			
	<u>As-Received</u>	<u>Hydrazine-Treated</u>	<u>n-Butylamine-Treated</u>	<u>Benzene-Treated</u>
C	0.15	0.2	0.3	0.2
H	9.92	9.78	9.86	9.77
N	0.18	0.22	0.24	0.19
Li	0.23	0.20	0.22	0.22
Cl	0.18	<0.05	0.12	0.13
Mg	1.18	1.18	1.18	1.15
O <sup>a</sup>	0.33	0.78	1.58	1.74

<sup>a</sup>Analysis by neutron activation.

(C) Of the three pretreated hydride samples, the one treated with n-butylamine was the most chemically stable in a hydrazine environment. The elemental analysis of the benzene-treated sample was similar to the analysis after n-butylamine treatment, but the benzene-treated hydride showed more initial gassing in hydrazine. In contrast, the chloride content of the hydrazine-treated sample was below the detection limit of the analytical technique, but the hydride did not form a gel with good physical integrity. The anomalies that have arisen in these experiments indicated that more work is needed to determine the correlation between gel physical integrity and such factors as ionic contamination from the hydride, hydride chemical stability, and surface conditions of the hydride.

4. Heterogeneous Propellant Preparation (U)

(C) Various parameters of propellant preparation are being studied as time permits. During this period, the effect of the particle size of AlH<sub>3</sub>-1451 and a new mixing sequence was investigated.

a. Particle Size (U)

(C) A gelled propellant with 0.42% polyacrylamide and AlH<sub>3</sub>-1451 fines (200 mesh) was prepared to determine the effect of particle size on physical properties. The sample was prepared by premixing the dry hydride and gelling agent to which hydrazine was added with mixing. The gel formed in about three minutes. The cohesive and shear properties were good and the propellant was easily extruded through a syringe. After 24 hours, the yield stress was 1595 dynes/cm.<sup>2</sup>, then dropped to 1372 dynes/cm.<sup>2</sup>

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after 48 hours and remained constant for five days, at which time the yield stress was 1380 dynes/cm.<sup>2</sup>. The sample was not large enough to continue testing past five days or to determine the effect of particle size on apparent viscosity. This will be accomplished in the future when a larger sample of small crystal size AlH<sub>3</sub>-1451 becomes available. From this small sample, it was concluded that the smaller particle size gave a slight improvement in flow properties and an increase in yield stress over the standard AlH<sub>3</sub>-1451 (80-150  $\mu$ ).

b. Mixing (U)

(U) A new method of propellant preparation was investigated which involved dispersing the agent in hydrazine for a 5-15 minute period with a high shear stirrer, followed by a shaking action for several hours. The hydride was added after the high shear mixing. Initial results indicate that very uniform propellant mixture was obtained by this method. In the future, the physical properties of propellants prepared by this method will be compared with the low shear mixing currently used for larger preparations.

B. STABILITY STUDIES (U)

(C) It has been shown that the amount of initial gas evolution and the long-term stability of AlH<sub>3</sub>-1451/N<sub>2</sub>H<sub>4</sub> formulations are related to the water content of the hydrazine and surface condition of the hydride (1). Work has now been directed toward more complete characterization and selection of the type of AlH<sub>3</sub>-1451 for scale-up of the heterogeneous propellant system.

1. Lot to Lot Variation of Aluminum Hydride (U)

(C) Magnesium-doped, diphenylacetylene (DPA) treated AlH<sub>3</sub>-1451 is the most stable type of aluminum hydride known and is being produced at Dow's Texas Pilot Plant. It has been shown that the Mg-doped, DPA treated hydride has better long-term stability than unstabilized aluminum hydride in aluminum hydride/hydrazine formulations (1). The Mg-doping was found to considerably improve long-term stability of AlH<sub>3</sub>-1451/N<sub>2</sub>H<sub>4</sub> formulations, but DPA may only incrementally improve the stability, as it apparently dissolves in the hydrazine.

(C) Three additional lots of Mg-doped, DPA treated AlH<sub>3</sub>-1451 have recently been received from the Texas Pilot Plant. These samples have been evaluated both neat and in hydrazine. These results, combined with analysis of the samples, have been compared with data obtained from samples of the Mg-doped, DPA treated hydride (Lot 09236) used in earlier studies.

<sup>1</sup>(C) Dow Quarterly Report Nr. HZ-4Q-67, Contract Nr. AF 04(611)-11606, May 1967, AFRPL-TR-67-133.

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(C) The 60°C. stability of the three new samples, 11076A, 11216A, and 11256A, is shown in Figure 2 along with the stability of Lot 09236, also tested neat at the same temperature. All four samples required from 170 to 240 hours to reach 0.5% decomposition.

(C) The analysis of the four samples of Mg-doped, DPA treated  $\text{AlH}_3$ -1451 are shown in Table IV. No large differences in composition are apparent for these samples.

Table IV

(U) Analysis of Four Mg-Doped, DPA Treated  
Aluminum Hydride-1451 Samples

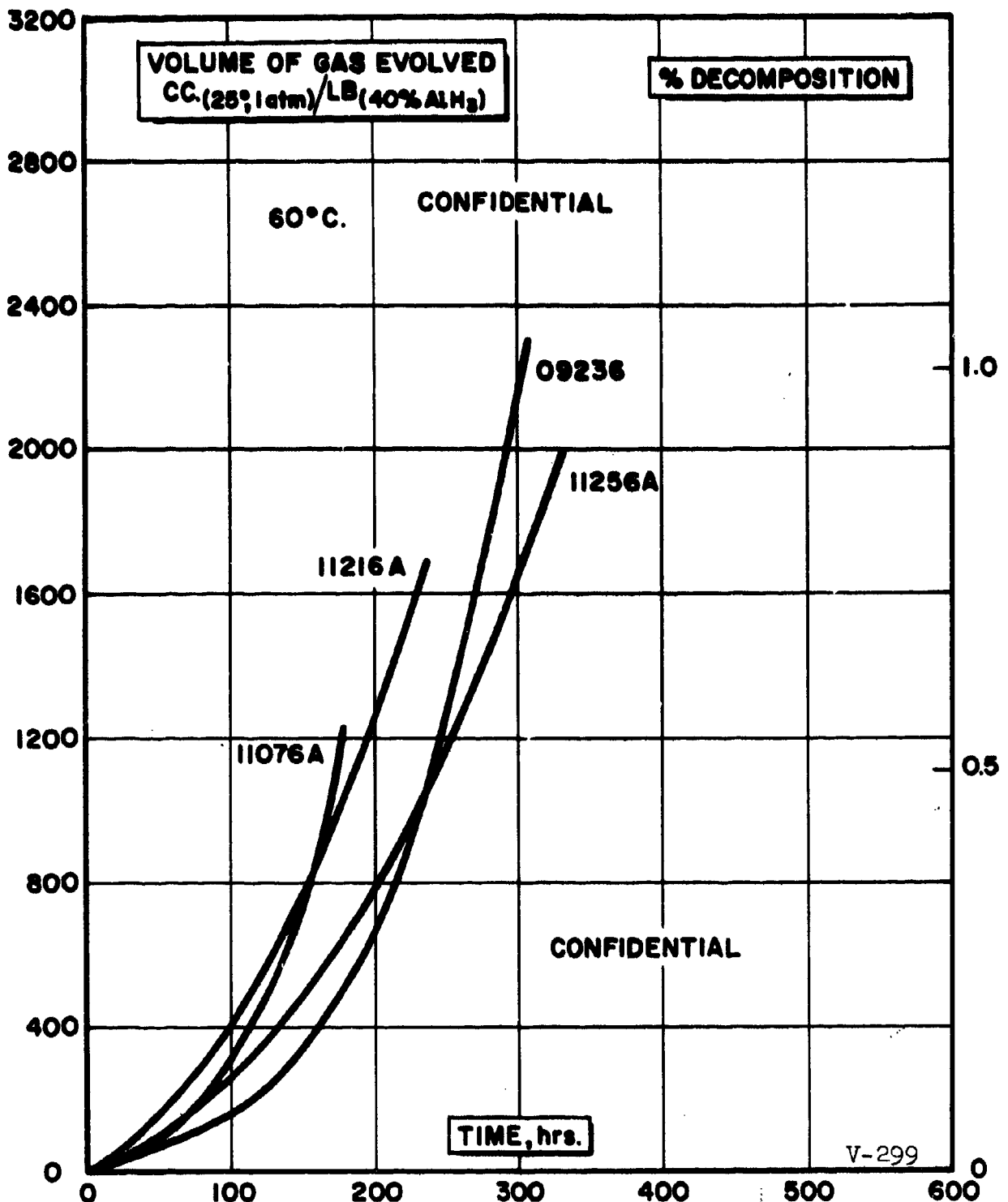
Lot No.	Bulk Dens. g./cc.	Elemental Analysis, %						
		Al	Cl	Li	C	H	Mg	O
11076A	0.84	87.8	0.07	0.18	0.70	9.90	1.11	--
11216A	0.78	87.4	0.10	0.21	0.88	10.05	1.16	0.48
11256A	0.79	87.8	0.10	0.18	1.01	9.96	1.16	0.42
09236	--	--	0.12	0.23	0.4	9.92	1.37	0.33

(C) The 60°C. stability of the four samples formulated at a 40% level in as-received hydrazine is shown in Figure 3. There is a significant difference in the amount of initial gas generated by the various samples of  $\text{AlH}_3$ -1451 when formulated in as-received hydrazine. There also appears to be a significant increase in the amount of initial gas from a sample of Lot 09236 hydride in hydrazine compared to an identical formulation prepared seven months previously. Re-examination of Lot 09236, which has been stored at -78°C., showed no change in its stability when tested neat at 60°C. Re-examination of the as-received hydrazine also showed no detectable change, although it is possible that the water content of the hydrazine has increased slightly. An increase in water content would explain the different results of the two stability tests.

(C) The behavior of the hydride-hydrazine formulation does not correlate with either the chemical composition or the stability of the neat hydride. The hydride/hydrazine formulation which has gassed the most contains the most stable hydride, Lot 11256A. Conversely, the least stable hydride, Lot 11076A, has produced the most stable hydride/hydrazine formulations. Also, the analytical data shown in Table IV do not show any clear indication of a relationship between the gas generation of  $\text{AlH}_3$ -1451/ $\text{N}_2\text{H}_4$  formulations and the amount of impurity in the hydride. Additional work is planned to define more clearly the differences between the various samples of hydride.

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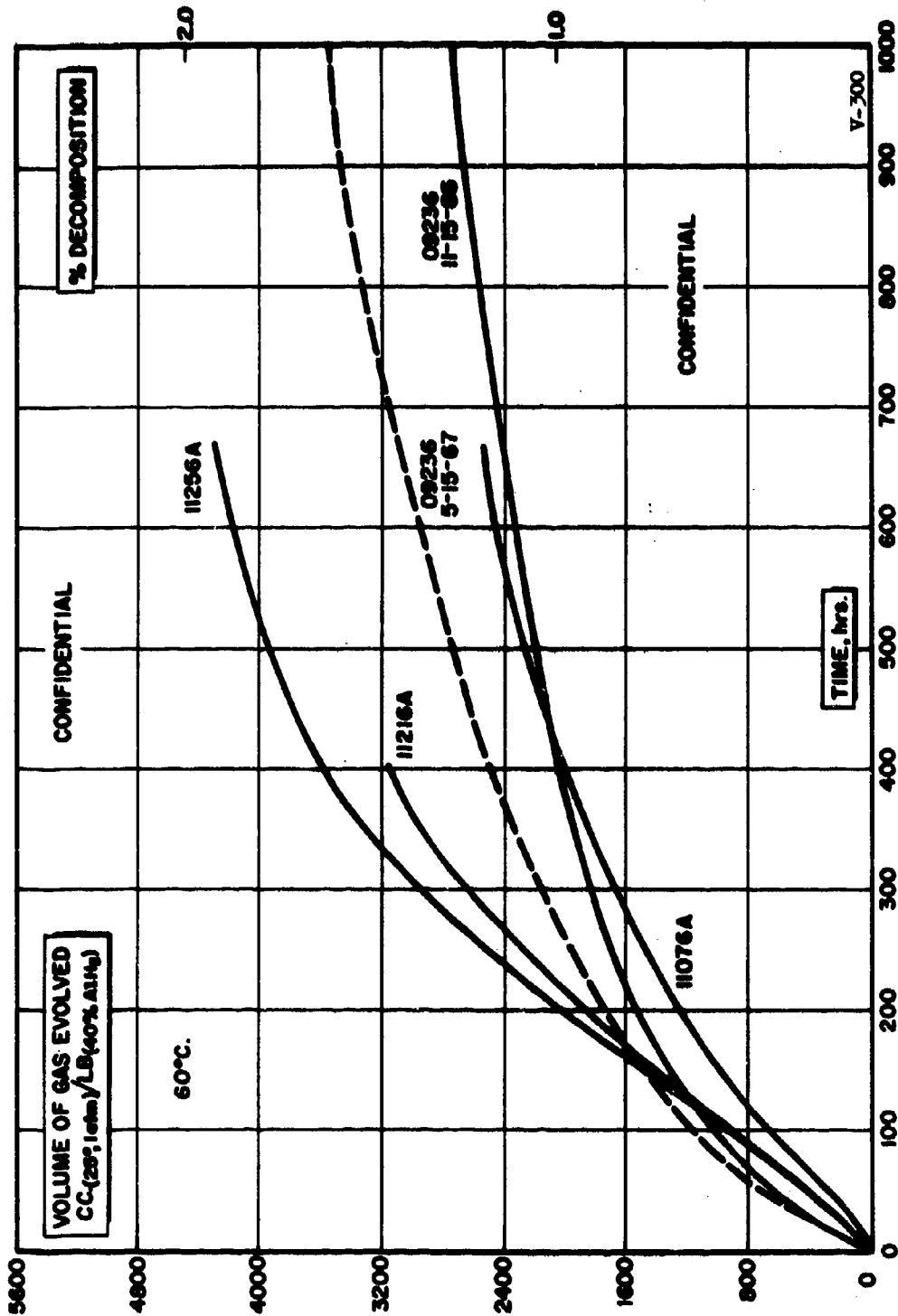


(C) Fig. 2 - 60°C. Stability of Four Mg-Doped, DPA Treated Aluminum Hydride-1451 Samples

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(C) Fig. 3 - 60°C. Stability of Four Mg-Doped DPA Treated Aluminum Hydride-1451 Lots in As-Received Hydrazine

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## 2. Finely Ground Aluminum Hydride (U)

(C) It has been shown that grinding  $\text{AlH}_3$ -1451 in the presence of  $\text{N}_2\text{H}_4$  creates some increase in the gassing rate (1). Additional work has been done to determine the effect of using small particle size hydride in the preparation of  $\text{AlH}_3$ -1451/hydrazine formulations.

(C) A sample of Lot 09236, Mg-doped, DPA treated  $\text{AlH}_3$ -1451, was finely ground and then formulated at a 40:60 ratio in as-received hydrazine. The gas generation rate of this sample was significantly greater than for an identical formulation containing unground hydride as shown in Figure 4. In addition, some of the ground hydride was tested neat at  $60^\circ\text{C}$ . and found to decompose faster than the unground material. It appears that the freshly exposed surface area of the hydride is important both in the neat hydride decomposition reaction and in the hydride-hydrazine initial gassing reaction.

## 3. Effect of Hydrazine on Autocatalytic Decomposition (U)

(C) In another experiment, neat, ground hydride was tested at  $60^\circ\text{C}$ . until it decomposed a total of 1%. It was then formulated in as-received hydrazine. The results shown in Figure 4 indicate the hydrazine effectively stopped the decomposition of the hydride. Surprisingly, the partially decomposed hydride produces less initial gas when formulated in hydrazine than the fresh hydride. This experiment suggests that  $\text{AlH}_3$ -1451, which is well into the autocatalytic portion of its decomposition, can be stabilized with hydrazine.

## 4. Ratio of Aluminum Hydride-1451 to Hydrazine (C)

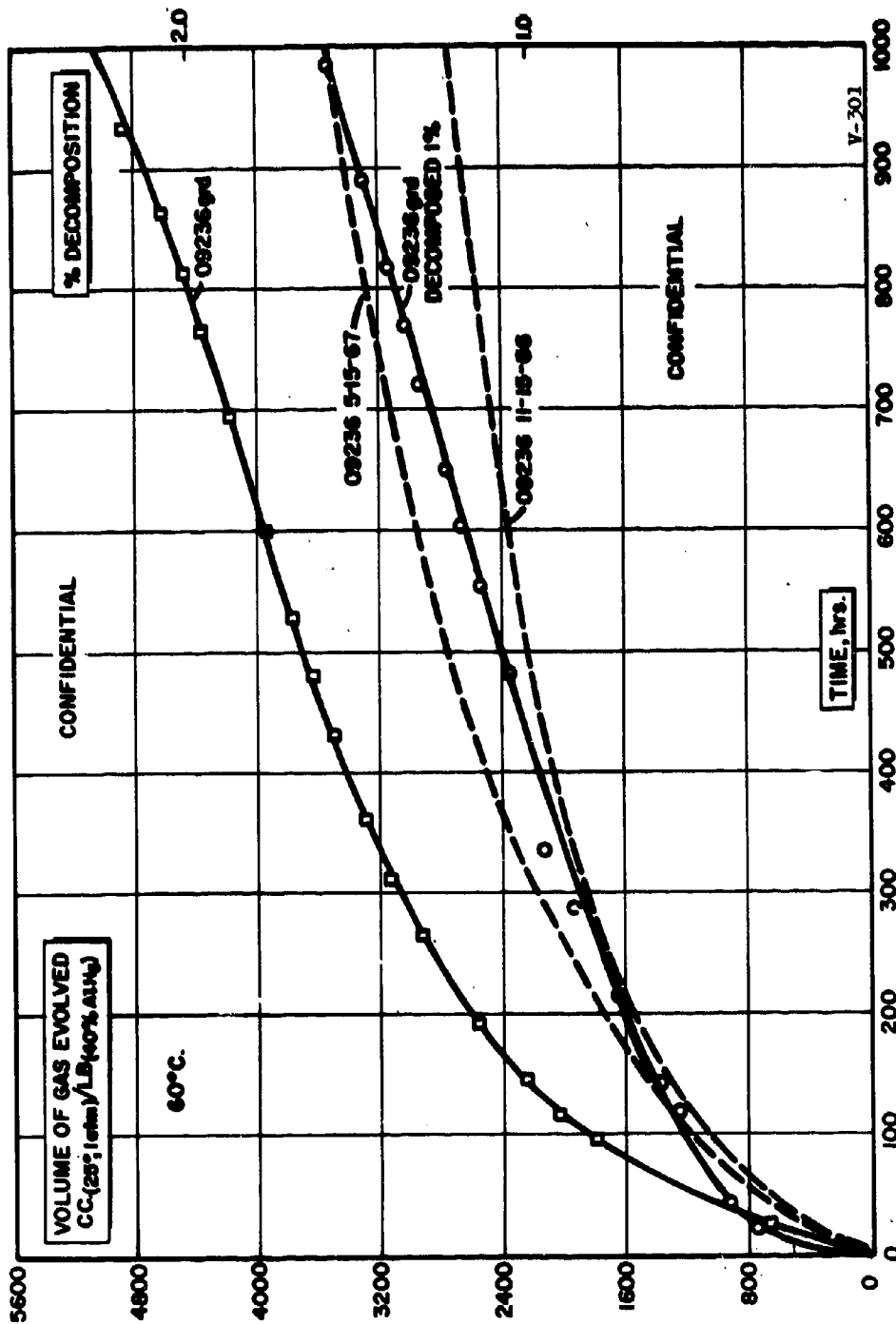
(C) Since it has been shown that 40%  $\text{AlH}_3$ -1451 can be contained in a gelled hydrazine propellant and since specific impulse considerations favor that concentration, most characterization of  $\text{AlH}_3$ -1451/ $\text{N}_2\text{H}_4$  propellant has been conducted at that loading. However, since the initial gassing reaction between the hydride and hydrazine may be dependent upon the ratio of the reactants, some useful information can be gained by studying various concentrations of hydride in the hydrazine.

(C) Formulations containing  $\text{AlH}_3$ -1451/ $\text{N}_2\text{H}_4$  in three different ratios, 1:2, 1:1, and 2:1, were prepared. The amount of gas evolved from these formulations is shown in Table V, based on the amount of hydride in the formulation, and in Table VI, based on the amount of hydrazine in the formulation. In Table V, the close agreement between the amount of gas evolved from the three formulations indicates the amount of gas is very dependent upon the amount of hydride in the formulations. Slightly less gas per unit of  $\text{AlH}_3$ -1451 is generated from the samples with lower concentrations of hydrazine, which indicates some dependence of the gassing reaction on the amount of hydrazine present. This dependence apparently increases with time.

<sup>1</sup>Ibid., HZ-4Q-67.

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(C) Fig. 4 - Gas Generation Studies of As-Received Ground, and Partially Decomposed Mg-Doped, DPA Treated Aluminum Hydride 1451 in Hydrazine

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Table V

(C) Gas Evolved From Aluminum Hydride-1451/Hydrazine Formulations at 60°C. Based on Amount of Aluminum Hydride-1451

Time, hr.	Gas Evolved, cc./lb. $\text{AlH}_3$ Ratio of $\text{AlH}_3$ -1451/ $\text{N}_2\text{H}_4$		
	1:2	1:1	2:1
17	821	801	788
41	1404	1366	1346
113	3232	2987	2579

Table VI

(C) Gas Evolved From Aluminum Hydride-1451/Hydrazine Formulations at 60°C. Based on Amount of Hydrazine

Time, hr.	Gas Evolved, cc./lb. $\text{N}_2\text{H}_4$ Ratio of $\text{AlH}_3$ -1451/ $\text{N}_2\text{H}_4$		
	1:2	1:1	2:1
17	411	801	1577
41	702	1366	2692
113	1616	2987	5158

(C) The data tabulated in Table VI indicate the amount of gas generated cannot be correlated to the amount of hydrazine present nearly as well as to the amount of hydride. The data in both Tables V and VI indicate the amount of hydride in hydrazine is the limiting reactant in the  $\text{AlH}_3$ -1451/ $\text{N}_2\text{H}_4$  initial gassing reaction. The amount of as-received hydrazine, at least in the range of 33% to 67%, is of secondary importance.

#### 5. Long-Term Stability at 25°C. (U)

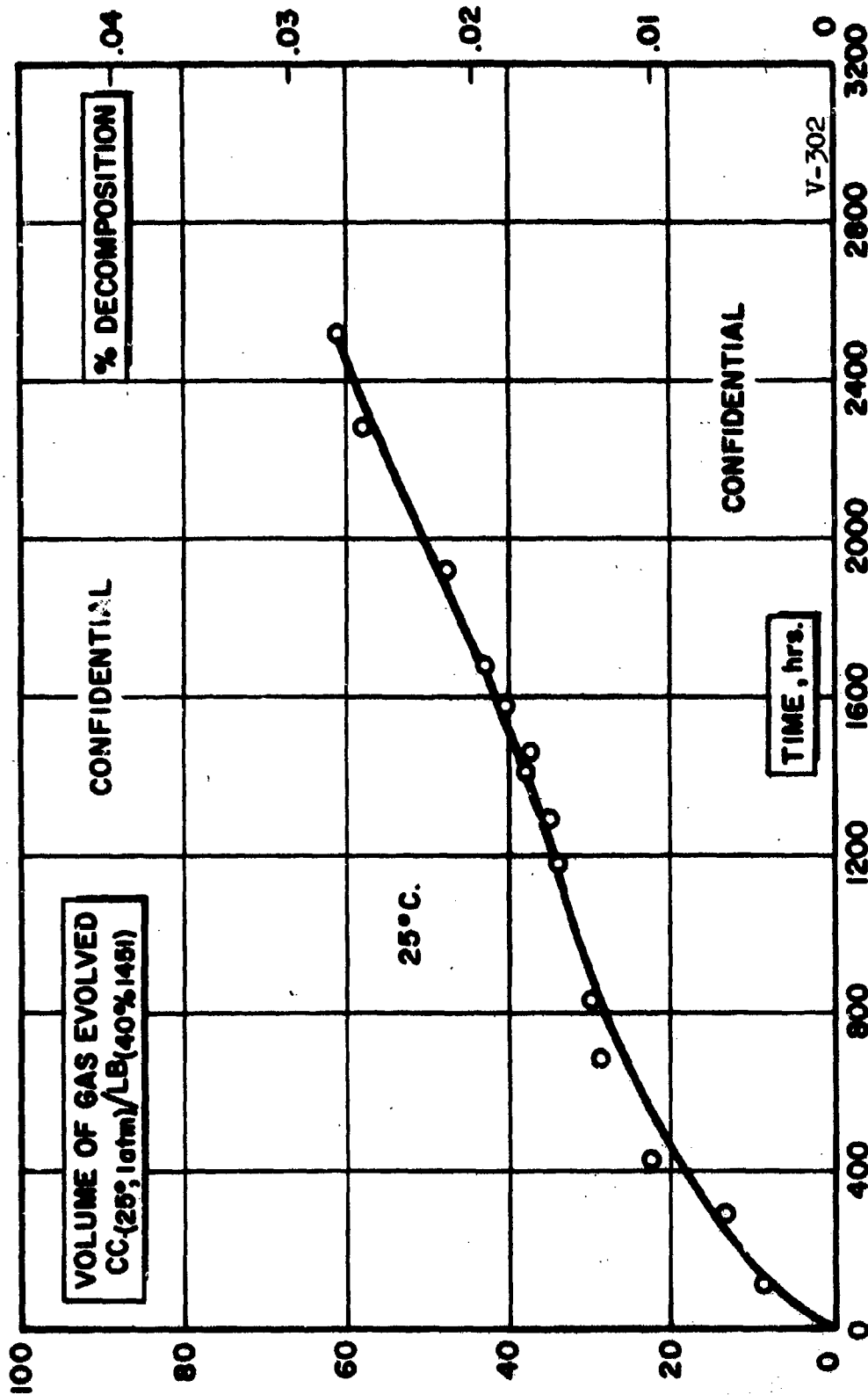
(C) The long-term stability of an  $\text{AlH}_3$ -1451/ $\text{N}_2\text{H}_4$  sample is still being measured at 25°C. The sample contains Mg-doped  $\text{AlH}_3$ -1451 and as-received hydrazine and had been held at 60°C. for a long period to complete the initial gassing reaction, as discussed in the last quarterly report (1).

(C) The data obtained from the 25°C. sample are shown in Figure 5. The gas generation rate at 2000 hours is  $3.5 \times 10^{-4}$  cc./min. Some temperature control problems are being encountered

<sup>1</sup>Ibid., HZ-4Q-67.

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(c) Fig. 5 - Long-Term Stability of an Aluminum Hydride-1451/Hydrazine Formulations at 25°C.

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during hot weather and may be responsible for the scatter of the data and the slight increase in gassing rate.

(C) The apparatus recommended by AFRPL for long-term surveillance of heterogeneous propellant has been constructed. A trial run will be started in the near future, but satisfactory pretreatment of the  $\text{AlH}_3$ -1451 will be required before meaningful long-term data can be obtained.

#### 6. Pretreatment (U)

(C) To reduce the initial gas evolution in  $\text{AlH}_3$ -1451/ $\text{N}_2\text{H}_4$  formulations, the  $\text{AlH}_3$ -1451 pretreatment studies were continued this quarter. The most effective  $\text{AlH}_3$ -1451 pretreatment has been in hydrazine (0.8%  $\text{H}_2\text{O}$ ) at  $60^\circ\text{C}$ . for greater than 100 days, as reported in the last quarterly report (1). The initial gas evolution was greatly reduced, but the treatment time is inconveniently long. Pretreatment parameters were studied with as-received hydrazine (0.8%  $\text{H}_2\text{O}$ ) to determine more precisely the factors that influence the reduction of initial gas evolution. Several treatment liquids were given precursory evaluations to find a system which would reduce the initial gas evolution as effectively as  $\text{N}_2\text{H}_4$ , but in a shorter treatment time. Ethyl alcohol and n-butylamine showed promise as treatment liquids. In the discussion which follows, all tests were with a hydride/treatment liquid ratio of 40:60, unless otherwise noted.

#### a. Hydrazine as Treatment Liquid (U)

(C) Several treatment parameters were varied in the hydrazine pretreatment procedure to determine their effect on amount of initial gassing that occurs when the hydride is reformulated with hydrazine. Pretreatment with temperatures greater than  $60^\circ\text{C}$ ., a greater proportion of  $\text{N}_2\text{H}_4$  in the treatment mixture, and water content of the hydrazine  $>0.8\%$  were performed to compare with the previous pretreatments.

#### (1) Treatment Temperatures (U)

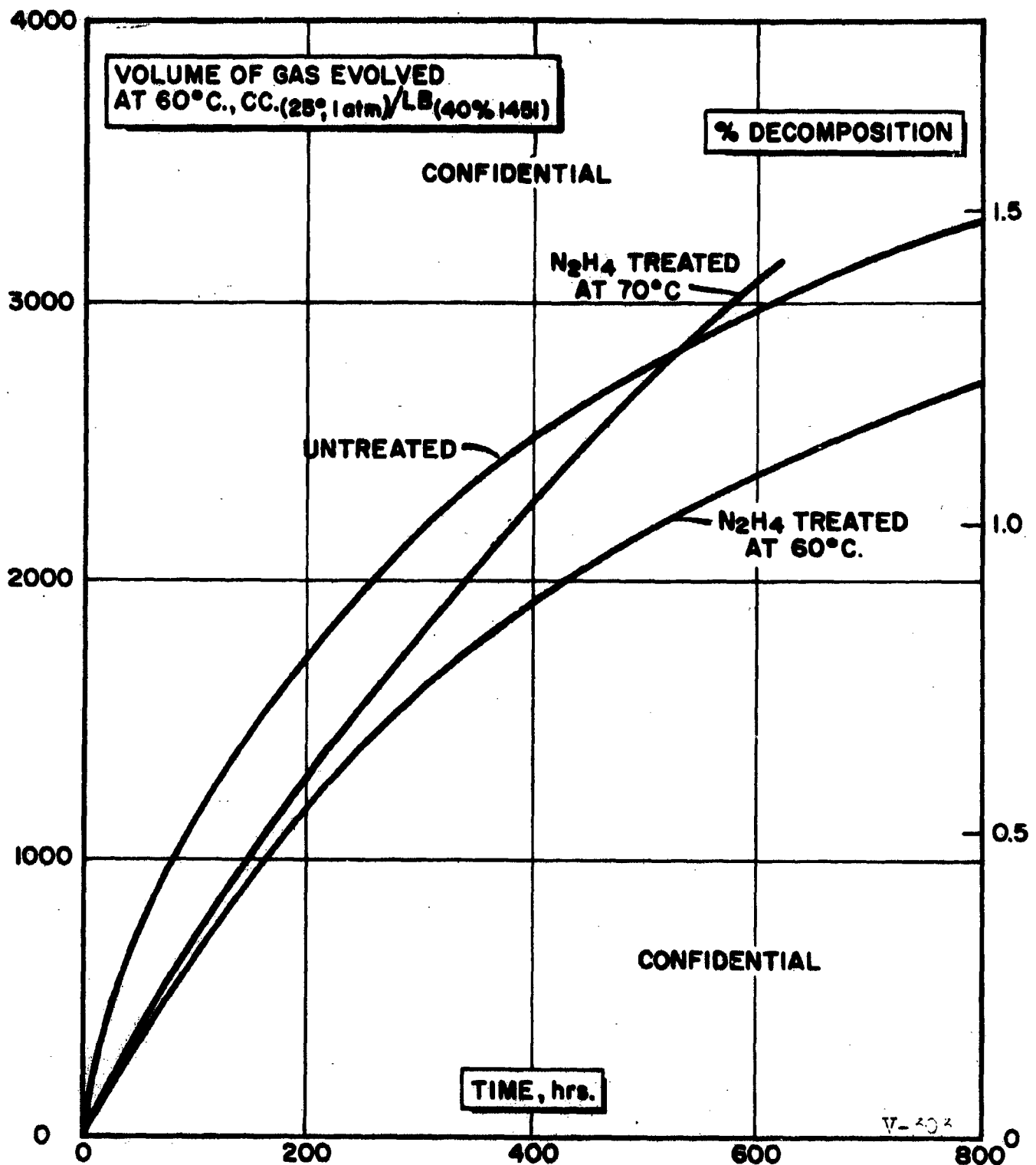
(C) As stated in the previous quarterly report (1), treatment of  $\text{AlH}_3$ -1451 with hydrazine at  $60^\circ\text{C}$ . stabilized the hydride more effectively than treatments at lower temperatures. To determine if the stabilization could be achieved in less time at temperatures greater than  $60^\circ\text{C}$ ., a sample of  $\text{AlH}_3$ -1451 was allowed to stand in contact with as-received hydrazine at  $70^\circ\text{C}$ . for six days. After recovery from the hydrazine and thorough drying, the hydride was reformulated with as-received hydrazine and placed in the  $60^\circ\text{C}$ . Taliani apparatus to measure the gas evolution. The stability data for the  $70^\circ\text{C}$ . pretreated hydride are shown in Figure 6 together with data from two reference samples of as-received and  $60^\circ\text{C}$ . pretreated hydride. The higher pretreatment temperature caused reduction of initial gassing. In addition, the oxygen content of the sample treated at  $70^\circ\text{C}$ . was 0.98%, as

<sup>1</sup> Ibid., HZ-4Q-67.



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(C) Fig. 6 - Gas Generation of Pretreated Aluminum Hydride-1451 (Lot 09236) in As-Received Hydrazine.

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compared to 0.78% after the 60°C. treatment. These results indicate that the pretreatments of  $\text{AlH}_3$ -1451 should be kept at approximately 60°C., since higher and lower temperatures have produced less effective stabilizations.

## (2) Water Content of Hydrazine (U)

(C) The amount of water in hydrazine has been shown in previous reports (1,2) to be proportional to the amount of initial gas evolution and to the degree of stabilization of  $\text{AlH}_3$ -1451/ $\text{N}_2\text{H}_4$  formulations. The correlation was based on experimental data with hydrazine containing 0.1-0.8% water. Most hydride pretreatments to reduce initial gas evolution had been with hydrazine containing 0.8% water. To determine if a greater amount of water would decrease initial gassing with shorter pretreatment time, hydrazine with 3.2% water was used in a six-day pretreatment experiment. The treated sample was recovered from the hydrazine, dried and reformulated as-received hydrazine. The sample was tested in the 60°C. Taliani apparatus. The results (see Figure 7) indicate there is no benefit in using increased concentrations of water.

(C) The hydride stability data from the hydrazine pretreatment are presented in Figure 7 along with benzene pretreatment stability data. Pretreatment with benzene containing either 0.8% or 3.2% water produces approximately the same stability when the hydride is formulated with as-received hydrazine. After benzene treatment with 0.8% water, the hydride contained 1.74% oxygen, and with 3.2% water the hydride contained 3.86% oxygen. On a long-term basis, compatibility of the system after the benzene treatment appears to be slightly better than after the hydrazine treatment, but the excessive amount of hydrolysis prevents the benzene treatment from being an attractive means of stabilizing the hydride.

## (3) Aluminum Hydride-1451/Hydrazine Ratio (C)

(C) The amount of as-received hydrazine in the pretreatment mixture was doubled, which increased the ratio of  $\text{AlH}_3$ -1451/ $\text{N}_2\text{H}_4$  from 1:1.5 to 1:3 and doubled the total amount of water in the mixture. If a specific amount of water was necessary at 60°C. to keep the stabilizing reaction proceeding at a practical rate, the greater amount of hydrazine should produce more stabilization in a given period of time.

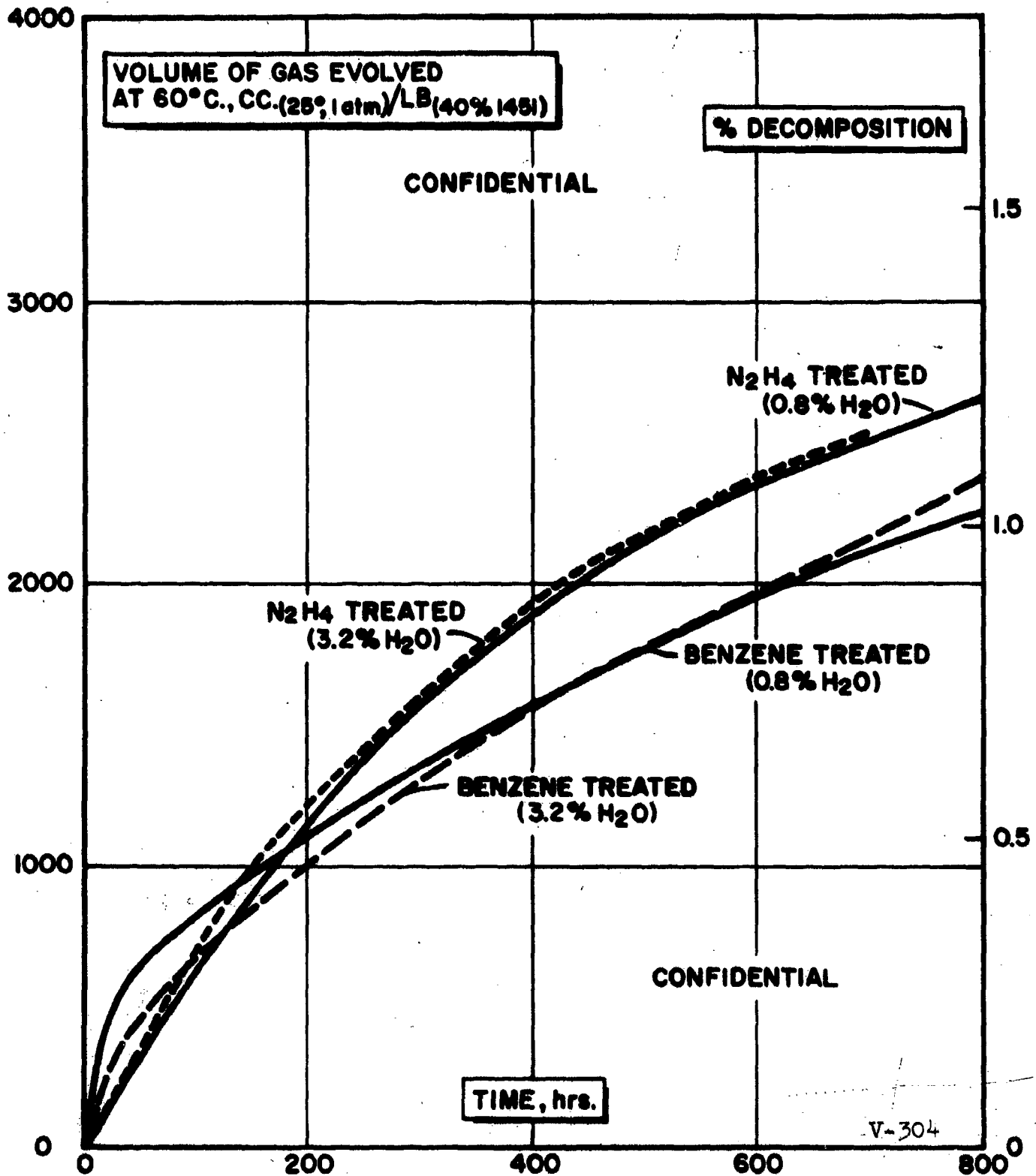
(C) The hydride was recovered after the 60°C. treatment in hydrazine, dried, and reformulated as-received hydrazine for the Taliani stability test. The stability data are presented in Figure 8 along with data from the 40:60 treatment ratio hydride and the untreated hydride. A slightly better reduction in initial

<sup>1</sup>Ibid., HZ-4Q-67.

<sup>2</sup>(C) Dow Quarterly Report Nr. HZ-3Q-67, Contract Nr. AF 04(611)-11606, February 1967, AFRPL-TR-67-43.

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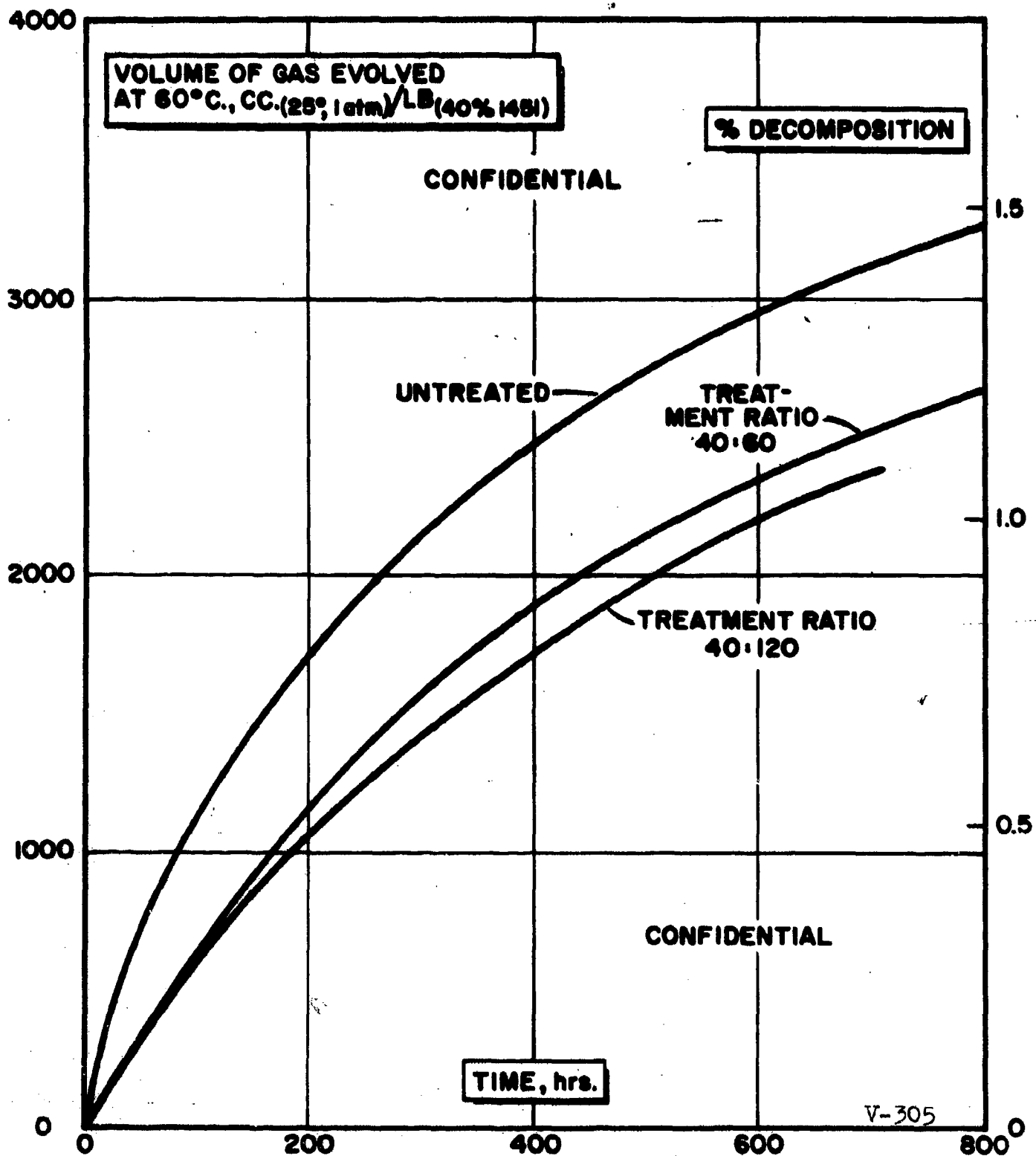


(C) Fig. 7 - Gas Generation of Pretreated Aluminum Hydride-1451 (Lot 09236) in As-Received Hydrazine.

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(C) Fig. 8 - Gas Generation of Pretreated Aluminum Hydride-1451 (Lot 09236) in As-Received Hydrazine

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gas evolution was achieved by using more hydrazine during the pre-treatment. The amount of hydrolysis also increased, as indicated by the oxygen analysis in Table VII.

Table VII

(C) Effect of Water Content on Aluminum Hydride-1451  
in Six-Day Hydrazine Treatment

<u>AlH<sub>3</sub>-1451/N<sub>2</sub>H<sub>4</sub></u> <u>Ratio</u>	<u>N<sub>2</sub>H<sub>4</sub> Water Content</u> <u>wt. %</u>	<u>Oxygen Content</u> <u>of AlH<sub>3</sub>-1451</u> <u>wt. %</u>
1:1.5	0.8	0.78
1:3	0.8	1.04
1:1.5	3.2	2.41

The slight increase in stability does not warrant the added expense of doubling the amount of pretreatment hydrazine.

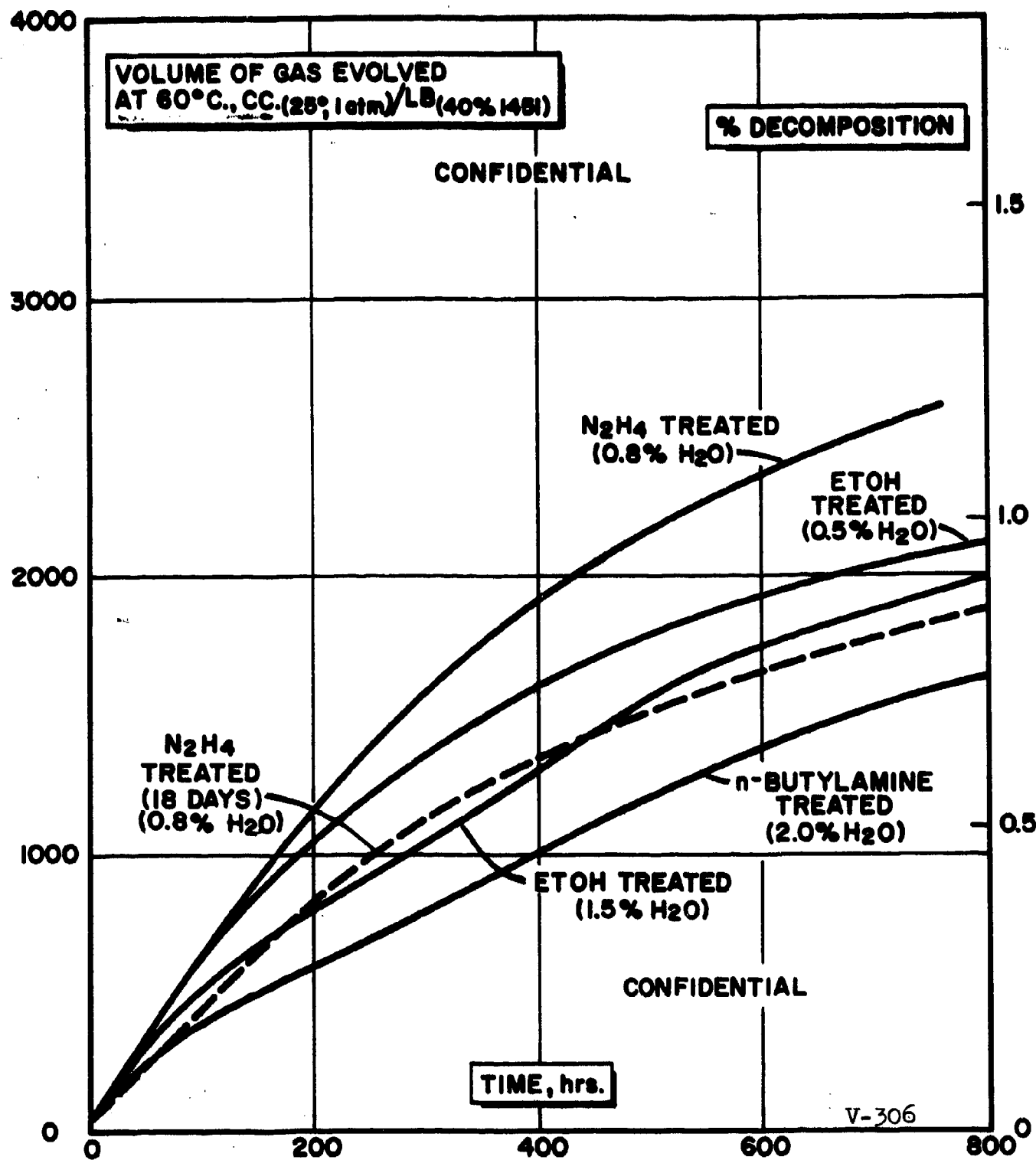
(4) Removal of Trace Ions (U)

(C) Elemental analyses of pretreated hydride have indicated that the hydrazine removes small amounts of lithium chloride from the hydride as well as improves its compatibility with hydrazine. The magnitude of lithium and chloride present in the samples was at the limit of detection for the analytical technique, so verification of analytical accuracy was desired. The hydrazine was collected from a hydride treatment and analyzed for lithium and chloride. Based on 5 g. of treated hydride, the 6.7 mmole of chloride found in the hydrazine represented 0.12 wt. %, which agreed well with the amount of lithium and chloride removed from the hydride (see Table III). Lithium and chloride were present in the hydrazine in essentially equal molar amounts, within the limits of the analytical technique, so the assumption that lithium chloride is being removed from the hydride is considered to be reasonably valid.

b. Other Treatment Liquids (U)

(C) In an effort to reduce both the time and cost of pre-treatment of AlH<sub>3</sub>-1451, a number of treatment liquids have been screened. The screening consisted of a six-day, 60°C. treatment of the hydride in the liquid, followed by recovery of the hydride, drying, and formulation with as-received hydrazine for stability testing on the Taliani apparatus. In all cases, the hydride/treatment liquid ratio was 40:60 on a weight basis.

(C) Of the treatment liquids tested, n-butylamine and ethyl alcohol have shown the most promise of reducing the initial gas evolution in less treatment time than hydrazine (see Figure 9).



(C) Fig. 9 - Gas Generation of Six-Day Pretreated Aluminum Hydride-1451 (Lot 09236) in As-Received Hydrazine

Tetrahydrofuran, n-propanol and ethyl acetate were not as effective. A six-day treatment with n-butylamine produced better stabilization than an eighteen-day treatment with hydrazine with a moderate increase in the hydride oxygen content (see Table VIII).

Table VIII

(U) Oxygen Analysis of Aluminum Hydride-1451  
Treated at 60°C. for Six Days

<u>Treatment</u> <u>Liquid</u>	<u>Water in Liquid</u> <u>Phase, wt. %</u>	<u>Oxygen Content of</u> <u>AlH<sub>3</sub>, wt. %</u>
n-Butylamine	2.0	1.58
Ethanol	0.5	0.87
Ethanol	1.5	1.30
Hydrazine	0.8	0.78
Hydrazine	0.8	1.22 <sup>a</sup>

<sup>a</sup>Eighteen-day treatment.

The alcohol treatment was intermediate in effectiveness between six-day treatments with hydrazine and n-butylamine.

(C) Ethyl acetate, n-propyl alcohol and tetrahydrofuran were screened as-received and were ineffective. All three liquids had a very low water content. Water will be added for future testing of these compounds as treatment media. Screening will also be continued on other amines as well as n-butylamine.

**SECTION II****(U) CONCLUSIONS AND FUTURE WORK****A. CONCLUSIONS** (U)

1. (U) A crosslinked polyacrylamide gellant was selected for further evaluation and system characterization in Phase II of the program.
2. (C) Lot to lot variations in Mg-doped, DPA treated  $\text{AlH}_3$ -1451 significantly affect the gas generation of  $\text{AlH}_3$ -1451/ $\text{N}_2\text{H}_4$  formulations.
3. (C) Differences in stability of  $\text{AlH}_3$ -1451/ $\text{N}_2\text{H}_4$  formulations do not correlate with differences in  $\text{AlH}_3$ -1451 stability or known composition.
4. (C) The polyacrylamide gelling agent is not detrimental to the stability of  $\text{AlH}_3$ -1451/ $\text{N}_2\text{H}_4$  formulations.
5. (C) Grinding the hydride prior to formulation is detrimental to the stability of  $\text{AlH}_3$ -1451/ $\text{N}_2\text{H}_4$  formulations.
6. (C) The effectiveness of the hydrazine pretreatment to decrease the initial gas evolution of  $\text{AlH}_3$ -1451/ $\text{N}_2\text{H}_4$  formulations has not been further improved by water content greater than 1.0%, larger proportion of hydrazine in the pretreatment mixture, and treatment temperature greater than 60°C.
7. (U) Trace amounts of LiCl are removed from the hydride by hydrazine pretreatment.
8. (C) With n-butylamine or ethyl alcohol as the pretreatment liquid, the initial gas evolution of the  $\text{AlH}_3$ -1451/ $\text{N}_2\text{H}_4$  formulations was decreased even more than with an equivalent hydrazine pretreatment.

**B. FUTURE WORK** (U)

1. (U) Phase II characterization of the selected system will be initiated.
2. (C) Long-term surveillance of heterogeneous  $\text{AlH}_3$ -1451/ $\text{N}_2\text{H}_4$  propellant will be conducted in the dilatometer apparatus recommended by AFRPL.
3. (U) Future characterization of various lots of  $\text{AlH}_3$ -1451 will be continued to obtain data to explain the differences in stability of  $\text{AlH}_3$ -1451/ $\text{N}_2\text{H}_4$  formulations.



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4. (U) The mode of aluminum hydride pretreatment will be varied to include a Soxhlet extraction and agitation during treatment.
5. (U) Other treatment liquids will also be screened, with emphasis on amines.
6. (U) Surveillance of polyacrylamide gelled propellant will be continued.
7. (U) The effect of treated hydride samples on propellant physical properties will be studied.

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13. ABSTRACT - Heterogeneous gels are being studied to develop a stable, storable IMH-1/hydrazine propellant. A polyacrylamide gelling agent was selected from candidate thickening agents, and a gelled propellant system was developed in Phase I. This system will be further evaluated and characterized in Phase II. The polyacrylamide system was chosen for its superior physical stability, since the physical properties of all the candidate systems satisfied the minimum requirements.  The chemical nature and stability of the system components are still being investigated. Pretreatment in any one of several liquids stabilize IMH-1. When pretreated IMH-1 is combined with hydrazine, the amount of gas generated in the initial reaction is reduced and the long-term gas generation rate decelerates as rapidly as with the untreated IMH-1 in hydrazine. The polyacrylamide gelling agent slightly increases the amount of initial gassing of the gelled IMH-1/hydrazine propellant, but the long-term stability equals the stability of ungelled IMH-1/hydrazine.		

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